

**IN SITU ANOMALOUS SMALL ANGLE X-RAY SCATTERING OF  $\text{LiMn}_2\text{O}_4$  DURING ELECTROCHEMICAL DELITHIATION.** *Artur Braun, Environmental Energy Technology Division, Lawrence Berkeley National Laboratory, One Cyclotron Road, Berkeley, CA 94720, Fax: 510-486-7303, abraun@lbl.gov, Sönke Seifert, Chemistry Division, Argonne National Laboratory, and Elton J. Cairns, Chemical Engineering Department, University of California, Berkeley*

The nano- and microstructure of battery electrodes was traditionally studied with electron microscopy. Notwithstanding, this technique usually does not allow for the in-situ characterization of electrodes due to the limited mean free path of electrons in solids, compared to x-rays.

The utilization of tunable x-ray energies from synchrotron radiation allows for an element specific scattering analysis. We therefore have succeeded to separate the scattering of the manganese from the scattering contributions of other electrode ingredients, such as carbon, electrolyte, and cell components.

We present data of the anomalous small angle scattering of manganese in the spinel  $\text{LiMn}_2\text{O}_4$  during electrochemical delithiation in an electrochemical in-situ cell. Within the  $Q$ -range of  $0.01 \text{ \AA}^{-1} < Q < 0.25 \text{ \AA}^{-1}$ , an oligomodal distribution of manganese-containing objects with gyration radii of about  $10 \text{ \AA}$ ,  $25 \text{ \AA}$  and  $80 \text{ \AA}$  was found ( see Figure ).

Structural reversible and irreversible changes were monitored during delithiation and subsequent lithiation.

Possible applications for the study of similar systems which exhibit colossal magnetoresistance and mechanisms of phase separations will be discussed also.

Figure: Scattering curve of the manganese in the electrode in the charged (delithiated) state, recorded at an x-ray energy of 6530 eV. Measured data are represented by dots. The drawn line is the sum of three least square Guinier fits to the data, after subtraction of incoherent background scattering and correction for the anomalous scattering.

